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CALIFORNIA INSTITUTE OF TECHNOLOGY
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Preface

This report is a quarterly summary of the research activities in the field of nondestructive testing of solid-propellant rocket motors, by the Jet Propulsion Laboratory.

QSR 38-1 presents an introduction to the problems and objectives of the program. Section I of this report details the progress in specific areas of research; Section II presents a continuing technical literature search of the subject.

The work reported herein is undertaken in partial fulfillment of the Advanced Research Projects Agency Letter Order No. 107-60, for the National Aeronautics and Space Administration.

W. H. Pickering, *Director*
Jet Propulsion Laboratory

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I. Physical Properties Studies

A. Effects of Moisture on Mechanical Properties

It was shown previously (*QSR 38-1*) that the mechanical properties of both polysulfide and polyurethane propellants are severely affected by moisture content of the surrounding air during storage at ambient temperature. The aging of polysulfide propellants has now been extended to 99 days, and the mechanism of moisture penetration into polyurethane propellants has been studied as follows:

(1) The effects of moisture on the properties of a dummy propellant and of two resin binders were ascertained.

(2) The amount of water absorption was determined quantitatively (weight) as a function of storage time at various humidities.

(3) Reversibility of mechanical properties was measured during storage in a dry environment after storage at various relative humidities.

The propellant compositions and the experimental procedure were described in *QSR 38-1*.

1. Composition of Dummy Propellant and Resins

The dummy propellant contained by weight 77 percent of Superbrite glass beads No. 149;[a] its binder was similar to that used in X-500-1 propellant, except that the cross-linker concentration was increased from 0.10 to 0.15 moles/mole of polypropylene glycol. The volumetric loading of filler in the dummy batch was much lower than in ammonium perchlorate propellants to permit casting, but initial mechanical properties were very similar to those of X-535 propellant.

The resins used in this study were identical with X-500 and X-535 binders. They were prepared with reduced

[a]Minnesota Mining and Manufacturing Co. Approximate size range: 0.0035 inch diameter and finer.

concentration of ferric acetylacetonate catalyst and cured in the form of sheets in both closed and open molds. Dumbbell specimens with a test section 0.25 inch wide and 1.0 inch long were die-cut from these sheets and tested at a jaw separation rate of 5 in./min (approximate strain rate 2.8/min).

2. *Polysulfide Propellants*

Additional storage data only confirmed the conclusions discussed in *QSR No. 38-1* and thus are not presented. It was noted, however, that both the tensile strength and the elongation continue to increase slowly over the entire 99-day aging period at low and intermediate humidities,

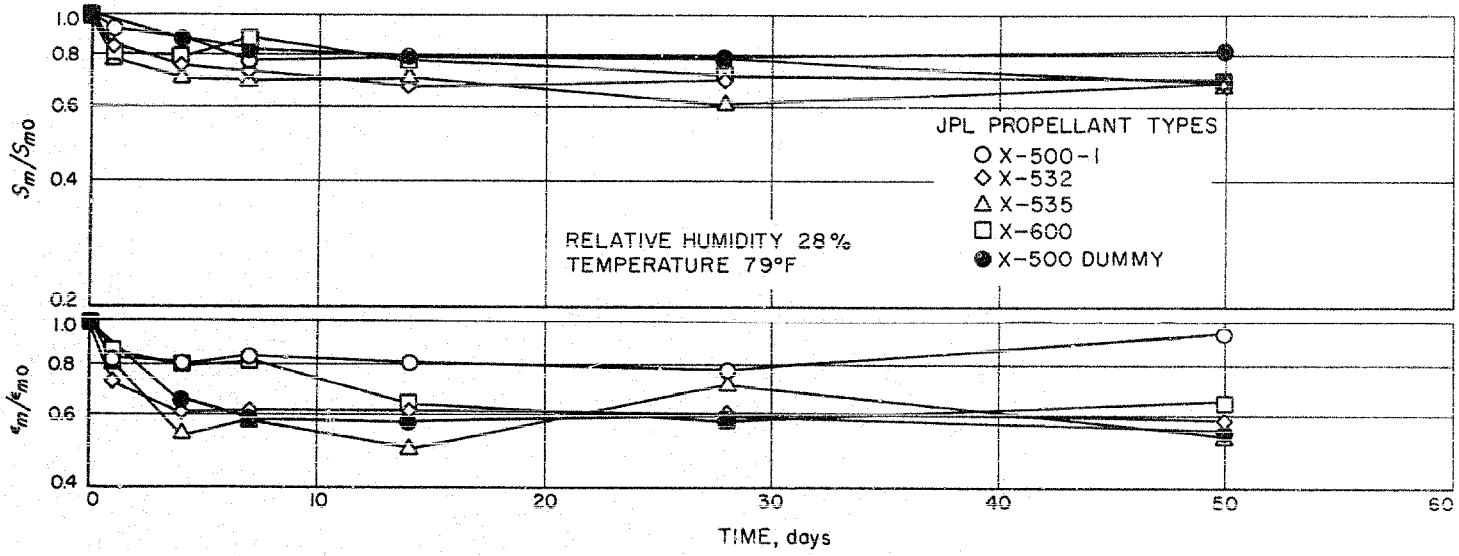

Figure 1. Changes in mechanical properties of polyurethane compositions during ambient aging at 28% relative humidity

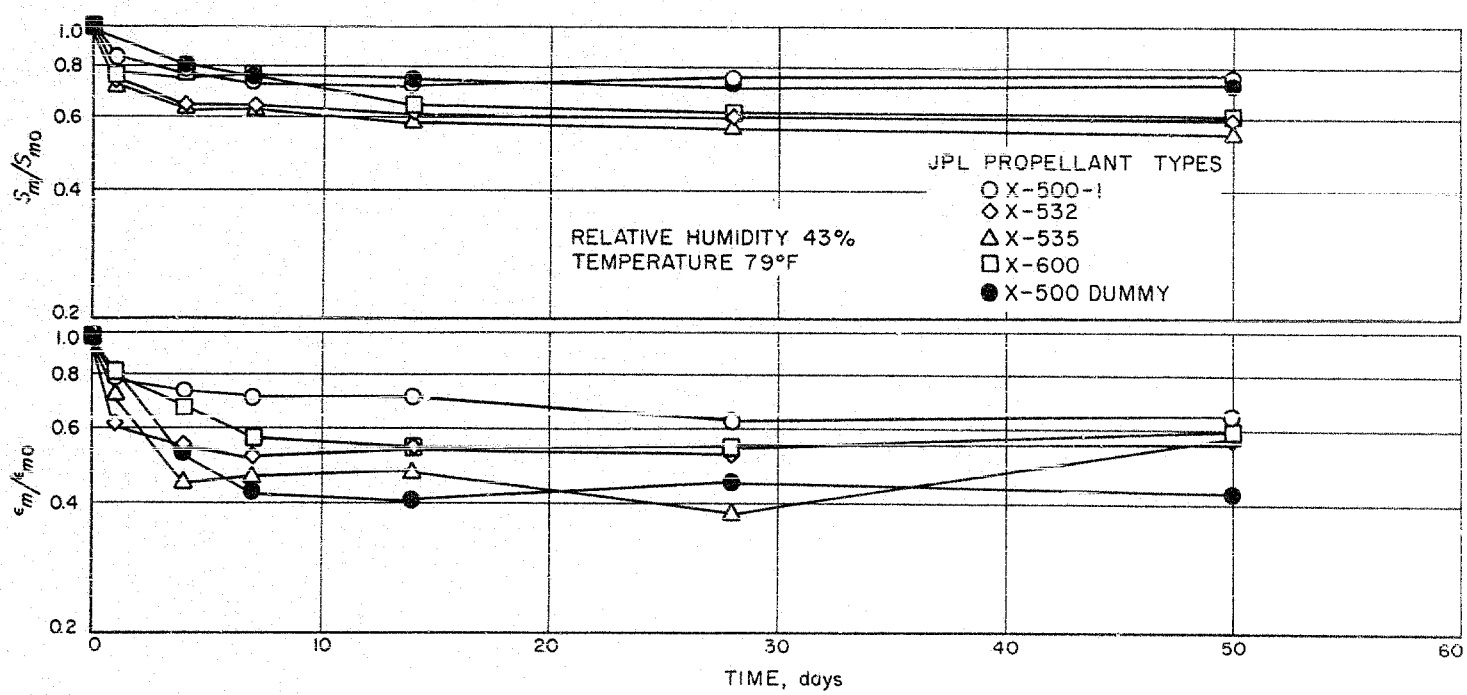

Figure 2. Changes in mechanical properties of polyurethane compositions during ambient aging at 43% relative humidity

even though near-equilibrium values are reached in 1 to 2 weeks.

3. Polyurethane Compositions

a. Effects of aging on tensile properties. The ratio of aged to initial tensile properties for a dummy composition containing glass beads is compared with data for polyurethane propellants in Figures 1–4. Three humidities selected for this comparison are different from those presented before (*QSR 38-1*), and data for 100 percent humidity are repeated.

As previously noted, the dummy batch was prepared with a resin binder similar in composition to X-500 propellant, but its initial mechanical properties more closely resembled those of X-535 propellant. This difference makes a quantitative comparison with propellants difficult. Nevertheless, it is seen that exposure to relative humidities below 70 percent affects the mechanical properties of the dummy composition about as much as it affects those of propellants.

Since ammonium perchlorate is not hygroscopic under these conditions (Ref 1), it does not contribute to the observed decrease in tensile strength and elongation. In a saturated atmosphere, however, the tensile strength of the glass bead composition drops rapidly and then remains constant, while the tensile strength of propellants continues to decrease. Thus, the nature of the filler does affect the mechanism of water absorption in a saturated atmosphere; this is entirely logical since the deliquescence point of ammonium perchlorate occurs at about 95 percent relative humidity (Refs 1 and 2). However, the dummy material shows a greater decrease in elongation than propellants, as shown in Figure 4. This result parallels the anomalous weight increase of the dummy composition (see the following) and cannot be reasonably explained at the present time.

b. Moisture absorption. The amount of moisture absorption into polyurethane compositions during ambient storage at six humidities is shown in Figures 5 and 6. The percent increase in weight is based on the binder content. At relative humidities of 65 percent or lower, the glass bead composition absorbs water at the same rate and to the same extent as the unfilled resin, within experimental error. Data for an equivalent X-500 propellant are unfortunately not available; the X-535 propellant,

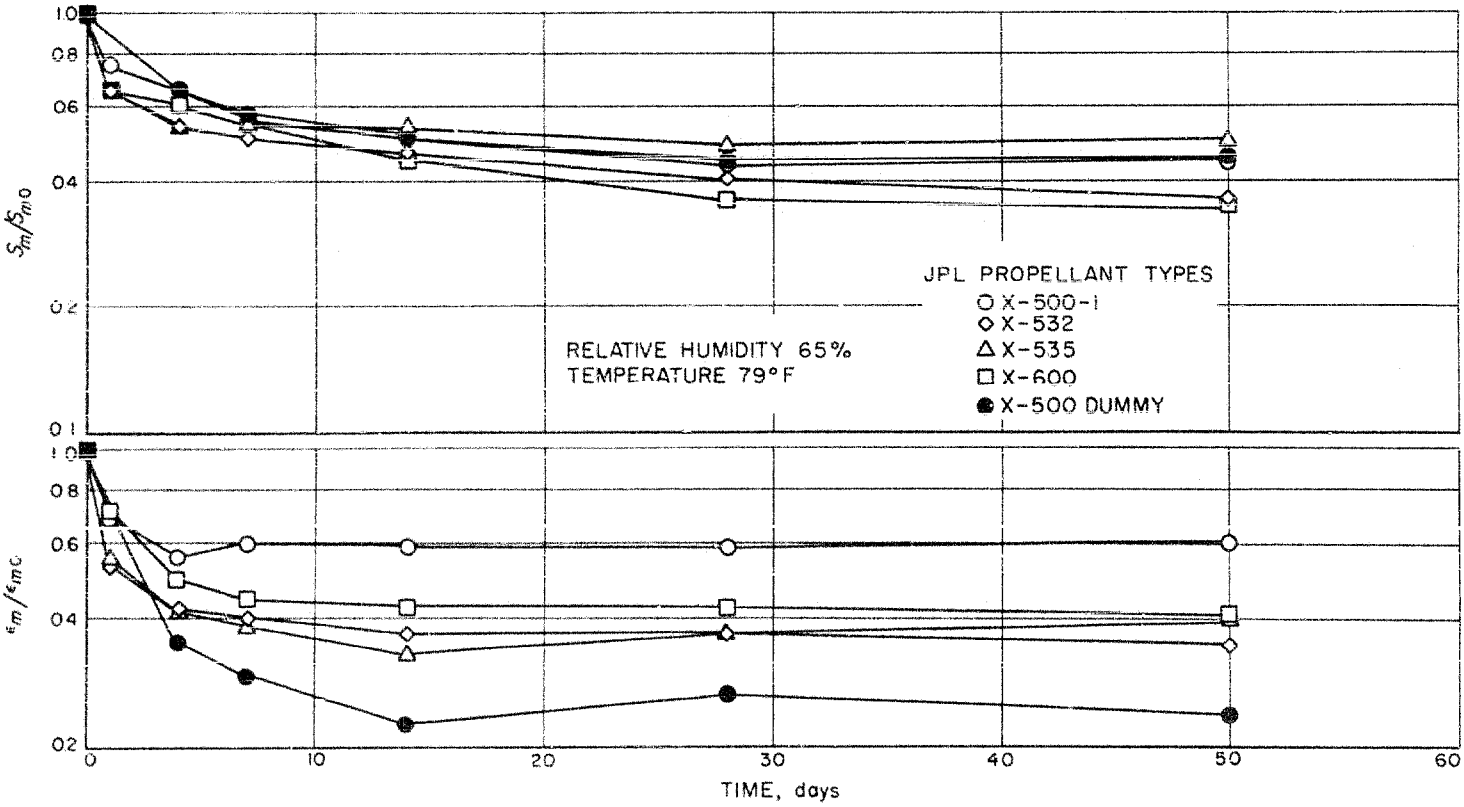

Figure 3. Changes in mechanical properties of polyurethane compositions during ambient aging at 65% relative humidity

which has a different binder composition and contains a wetting agent, does absorb water at a somewhat greater rate. Nevertheless, the difference is rather small, and the results strongly suggest that water absorption is independent of the filler and is primarily a function of the binder composition. In a saturated atmosphere, the filled compositions absorb much more water than the resin alone. This would be expected for the propellant containing ammonium perchlorate; the reason for continuing water absorption into the glass-bead filled composition is not known.

c. Reversibility of mechanical properties. To evaluate the recovery of mechanical properties, weighed specimens of X-535 propellant were aged at various humidities for 14, 28, and 50 days. At the completion of each storage

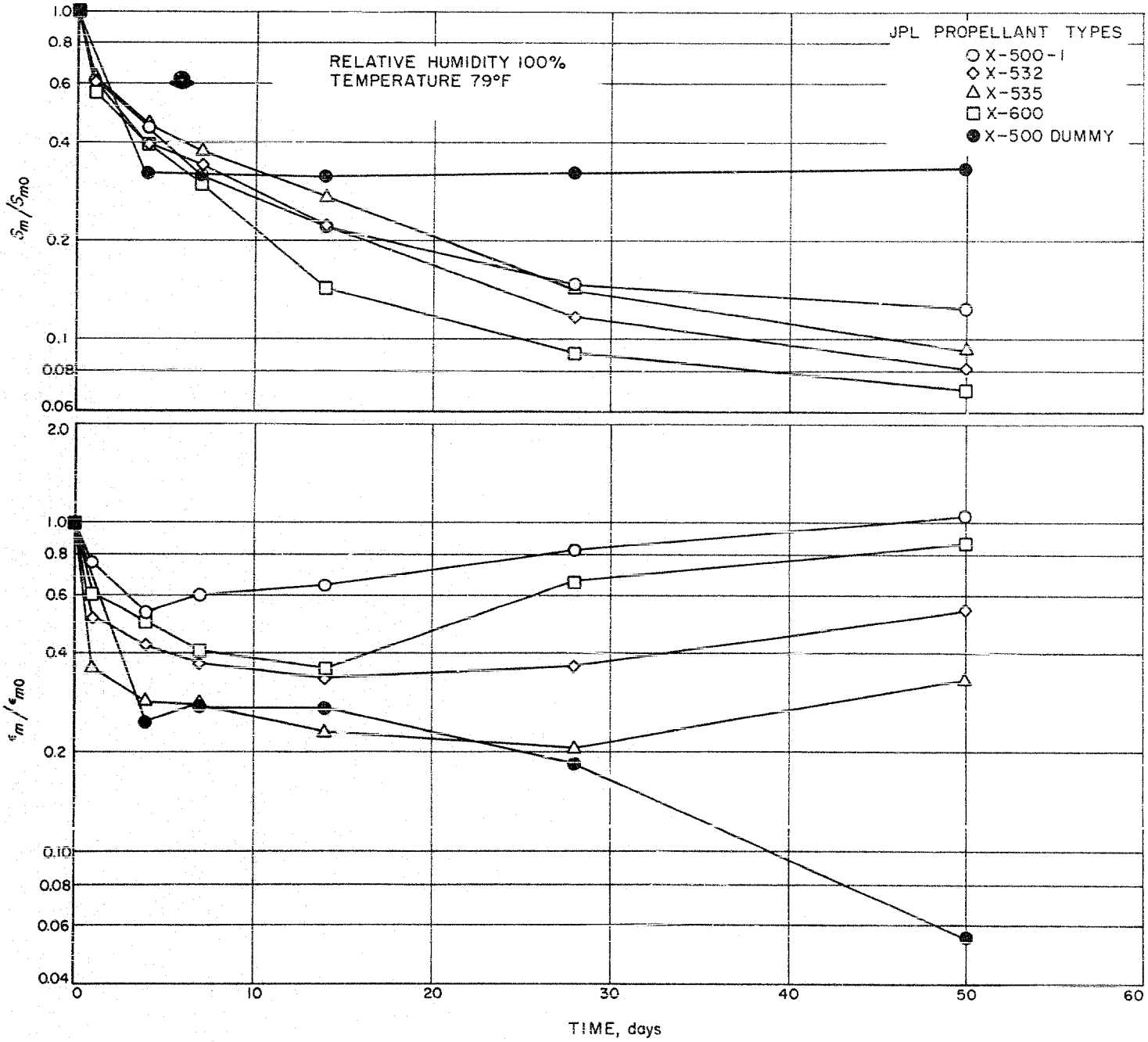

Figure 4. Changes in mechanical properties of polyurethane compositions during ambient aging at 100% relative humidity

period, two specimens were reweighed and tested, and the remaining were transferred into a dry atmosphere and allowed to recover at 79°F for varying lengths of time. Periodically, samples were withdrawn, weighed, and tensile properties were measured.

The changes in weight and in mechanical properties, observed after exposure to humidities of 65 percent or less, are substantially reversible, as illustrated in Figure 7. Samples of X-535 propellant aged for 14 days at various humidities increased in weight, and their tensile strength and elongation decreased severely. On transfer to a dry atmosphere at ambient temperature, water was removed and mechanical properties recovered rapidly, reaching 90 percent or better of their original value after 4 weeks of drying. Similar recovery was achieved after exposure to various humidities for 28 or 50 days. This result confirms that the observed deterioration of mechanical properties is physical in nature and that no chemical degradation of the binder takes place.

4. Conclusions

Surprisingly, samples of unfilled resin binders stored at various humidities absorb water, but do not change appreciably in mechanical properties. Thus, it is most likely that moisture absorbed into propellants does not change the properties of the binder, but disrupts the adhesive forces between oxidizer and binder. This destroys the reinforcing effect of the filler and causes a severe decrease in tensile strength and elongation at maximum stress. When the water is removed, the bond

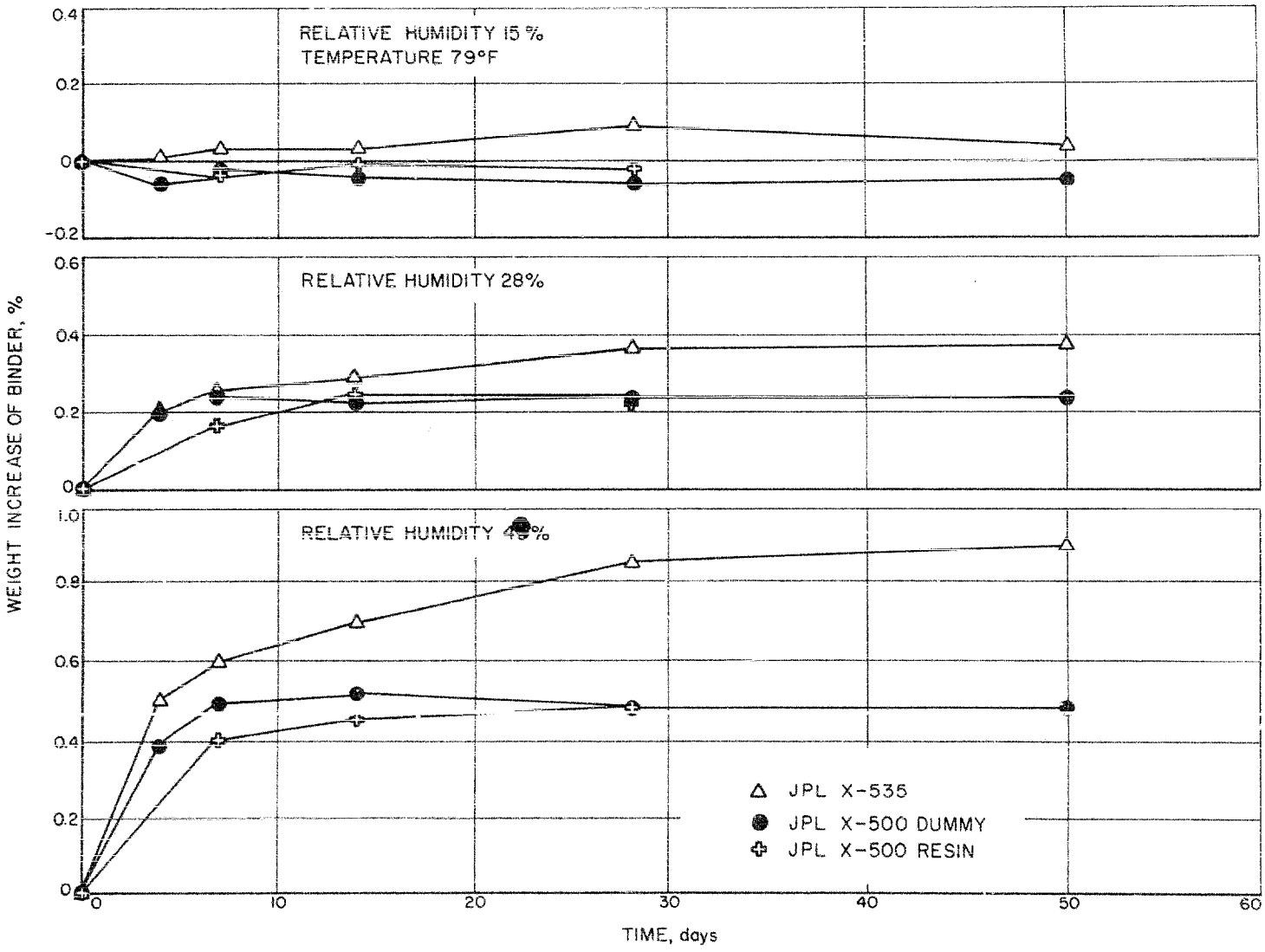

Figure 5. Weight increase of polyurethane systems during ambient aging at 15, 28, and 43% relative humidity

apparently reforms and the original mechanical properties are restored.

Storage in a saturated atmosphere, above the deliquescence point of ammonium perchlorate, presents a special case. Tensile specimens become saturated with water and, even though strain at maximum stress is very low, the stress frequently reaches a second maximum after an initial yield point and the ultimate strain is greatly increased, in agreement with previously reported results (Refs 3 and 4). After drying, the samples show a weight loss as indicated in Figure 7, reflecting some extraction of oxidizer. While about 90 percent of original tensile strength is recovered, the strain at maximum stress is increased by as much as 70 percent. The shape of the stress–strain curve is also altered, as shown in Figure 8. Thus it is apparent that exposure of propellant samples to saturated air causes permanent changes.

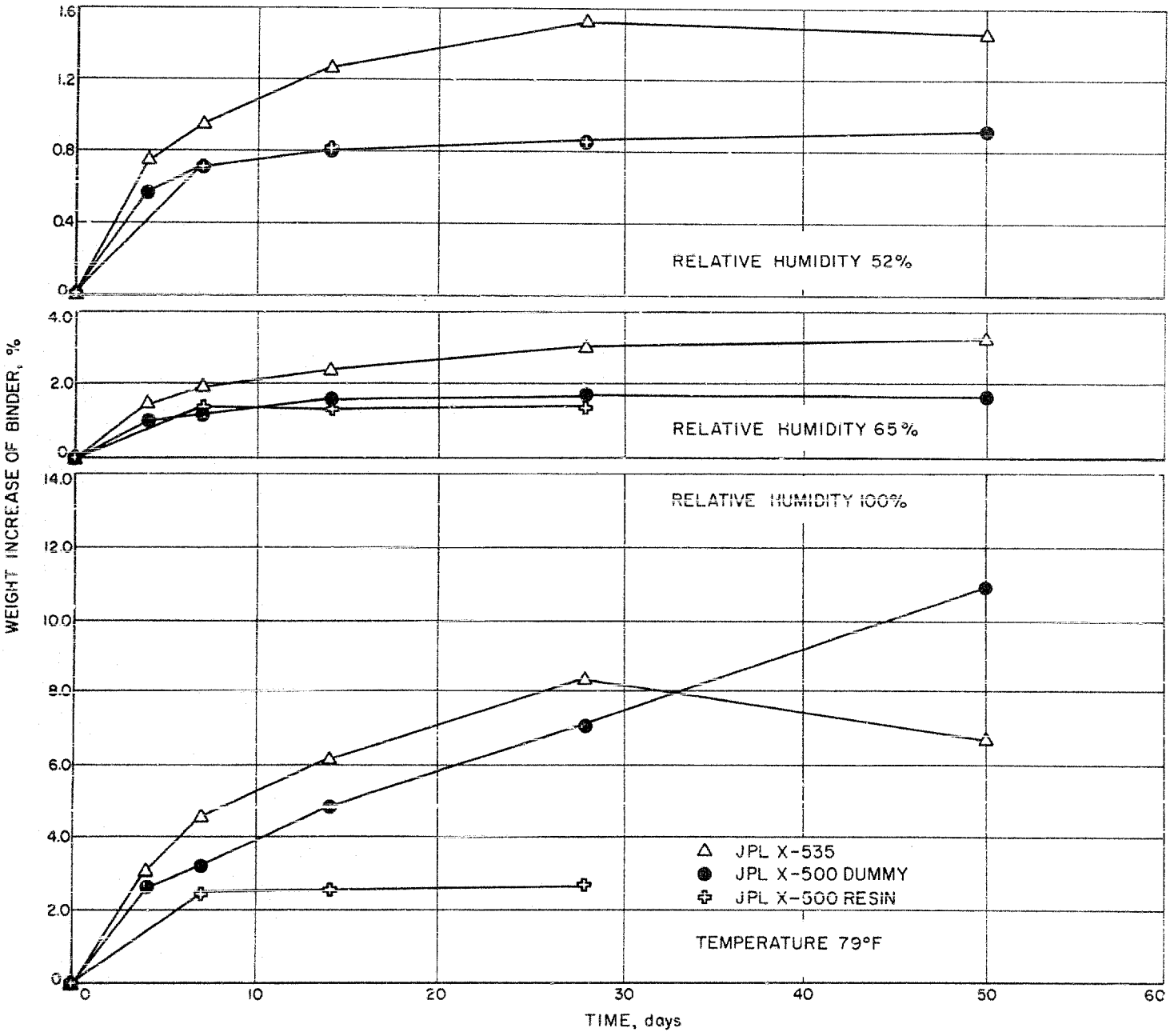

Figure 6. Weight increase of polyurethane systems during ambient aging at 52, 65, and 100% relative humidity

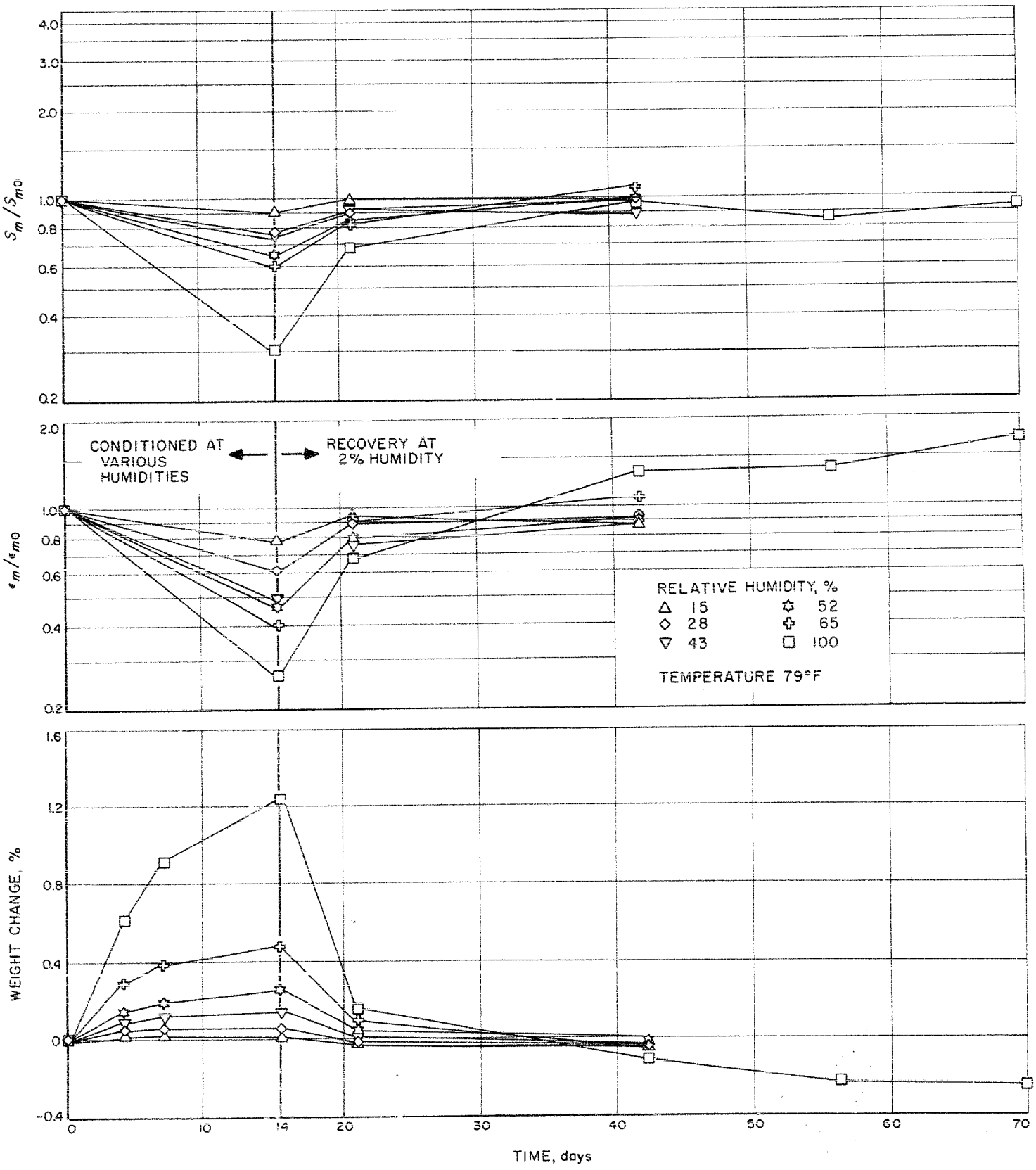

Figure 7. Changes in weight and in mechanical properties of JPL X-535 propellant during ambient aging at various humidities and during recovery at 2% relative humidity

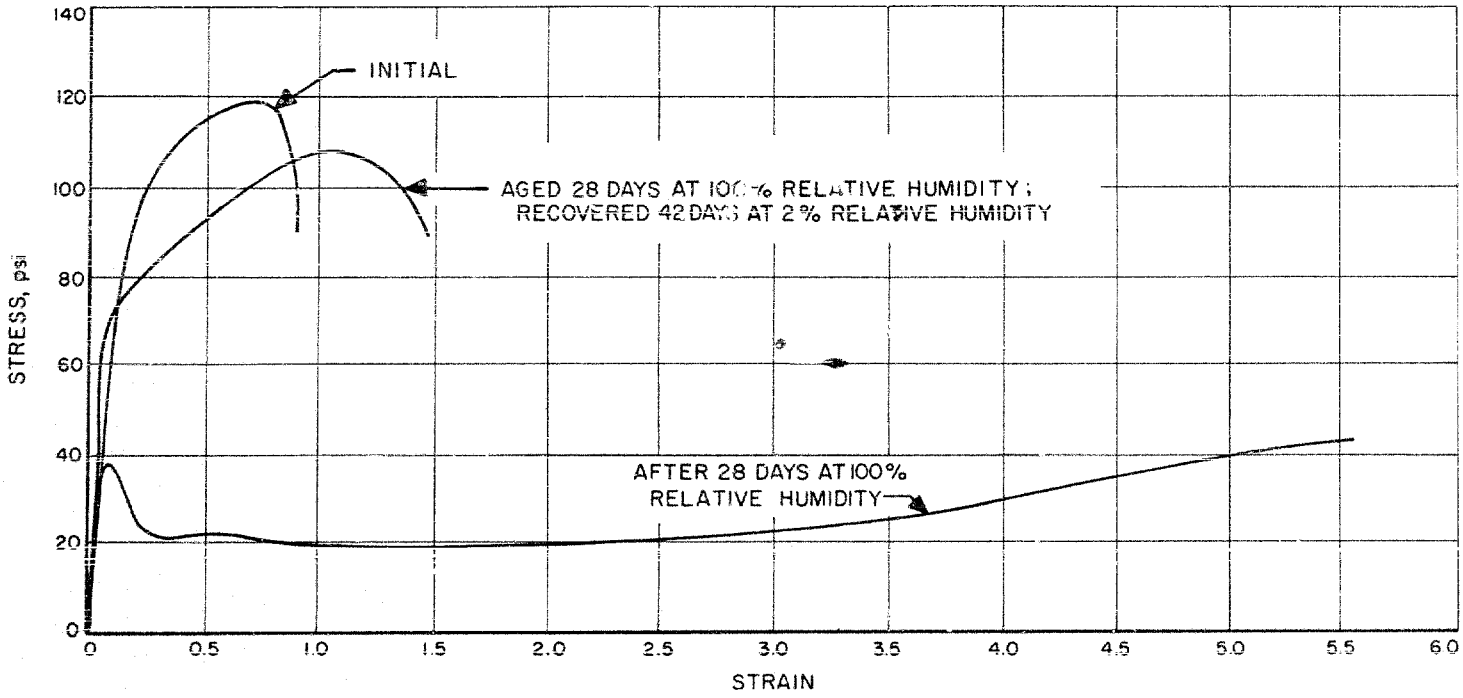

Figure 8. Typical stress–strain curves of X-535 propellant before and after ambient aging at 100% relative humidity, and after recovery at 2% relative humidity

B. Volume Changes Under Tensile Deformations

1. Calculation of Poisson's Ratio

Classical strain analyses define Poisson's ratio in terms of Cauchy strains as

$$\nu = -\frac{\varepsilon_2}{\varepsilon_1} \tag{1}$$

where ε_1 is the longitudinal strain in the direction of the applied stress and ε_2 is the lateral strain. In small-deformation elastic theory (neglecting second and third order effects), it is approximately true that ν is constant and equal to ½, if the material is incompressible. However, for finite strains such as occur in propellant grains, this is no longer true, and it can be easily shown that Poisson's ratio for an isotropic, incompressible material is a nonlinear function of the tensile strain given by

$$\nu = \frac{1}{\varepsilon_1}\left[1 - (1+\varepsilon_1)^{-\frac{1}{2}}\right] \tag{2}$$

If a volume change occurs on straining, it is convenient to express Poisson's ratio as a function of the principal extension ratio $\alpha \equiv \lambda_1 = 1 + \varepsilon_1$ and the volume ratio V/V_0. In order to do this, it is still necessary to assume that the material is isotropic and that the volume change is given by the relation

$$V/V_0 = (1+\varepsilon_1)(1+\varepsilon_2)^2 \tag{3}$$

Solving Equation 3 for ε_2 and substituting Equation 1, the expression for Poisson's ratio becomes

$$\nu = \frac{1}{\alpha - 1}\left[1 - \left(\frac{V}{V_0}\right)^{\frac{1}{2}} \alpha^{-\frac{1}{2}}\right] \tag{4}$$

For an incompressible material, $V/V_0 = 1$ and then Equation 4, of course, reduces to Equation 2. In this work, Equation 4 will always be used to calculate Poisson's ratio for propellants unless otherwise indicated.

Unfortunately, other definitions of Poisson's ratio are sometimes used for convenience in reducing and interpreting experimental results (e.g., Ref 5). Any definition used with Equation 3 will yield expressions analogous to Equations 2 and 4, but the results are numerically quite different. This is seen from calculations summarized in Table 1. In addition, the same three functions for incompressible materials are compared in Figure 9, showing vastly divergent results. This point is emphasized because numerical values obtained by one definition are sometimes quoted and used in strain analyses based on a

Table 1. Poisson's ratio as a function of α and V/V_0 for three definitions

Definition	Poisson's ratio, $\nu = f(\alpha, V/V_0)$	
	No volume change, $V/V_0 = 1$	Volume change, $V/V_0 \neq 1$
Classical Cauchy, $\nu_1 = -\frac{\varepsilon_2}{\varepsilon_1}$	$\nu_1 = \frac{1}{\alpha - 1}(1 - \alpha^{-1/2})$	$\nu_1 = \frac{1}{\alpha - 1}\left[1 - \left(\frac{V}{V_0}\right)^{1/2} \alpha^{-1/2}\right]$
Differential Cauchy, $\nu_2 = -\frac{d\varepsilon_2}{d\varepsilon_1}$	$\nu_2 = \frac{1}{2}\alpha^{-3/2}$	$\nu_2 = \frac{1}{2}\left(\frac{V}{V_0}\right)^{1/2} \alpha^{-3/2} - \frac{1}{2}\alpha^{-1/2}\left(\frac{V}{V_0}\right)^{-1/2} \frac{d\left(\frac{V}{V_0}\right)}{d\alpha}$
Differential Hencky, $\nu_3 = -\frac{d \ln(1 + \varepsilon_2)}{d \ln(1 + \varepsilon_1)}$	$\nu_3 = \frac{1}{2}$	$\nu_3 = \frac{1}{2} - \frac{1}{2}\frac{d \ln\left(\frac{V}{V_0}\right)}{d \ln \alpha}$

different definition of ν. Obviously, such a procedure makes any conclusions quite meaningless.

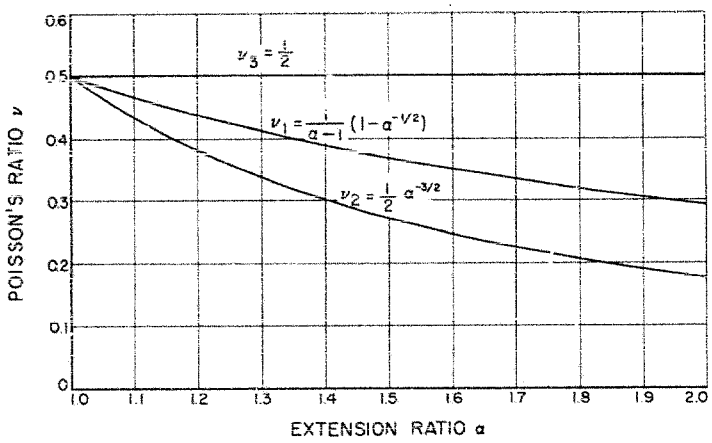

Figure 9. Poisson's ratio for incompressible materials as a function of α for three definitions (see Tab 1)

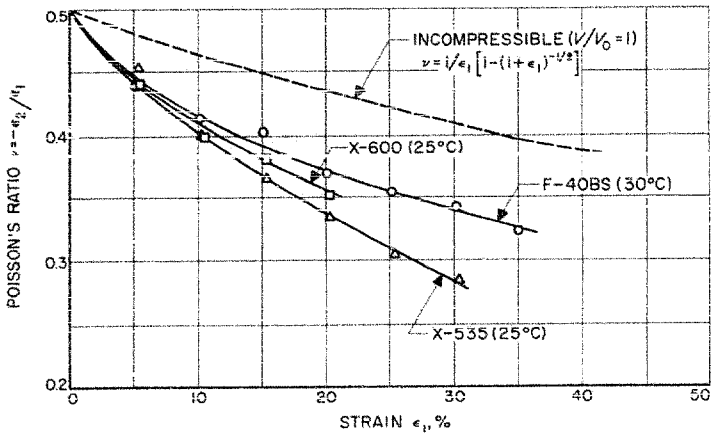

Figure 10. Poisson's ratio as a function of strain for three polyurethane propellants

2. Test Results

The data obtained thus far are insufficient to indicate the rate of volume increase and recovery as a function of time, though some interesting trends are becoming apparent. The results shown in Figure 10 (see Tab 2 for propellant compositions) represent the maximum volume

Table 2. Composition of polyurethane propellants[a]

Composition	Weight, %		
	F-40BS HU-1	X-535 S-101	X-600 PD-79
Ammonium perchlorate, unground	53.78	56.00	55.87
Ammonium perchlorate, ground	23.05	24.00	23.94
Lithium perchlorate	—	—	0.19
Aluminum MD-105	2.00	—	—
Copper chromite Cu 0202	1.50	—	—
Polypropylene glycol 2025	16.68	17.20	13.31
P-II polymer[b]	—	—	5.03
1,2,3-trimethylol propane	0.20	0.09	0.09
Alrosperse 11p[c]	0.48	0.50	—
Phenyl-β-naphtylamine	0.25	0.25	0.25
Ferric acetylacetonate	0.04	0.04	0.01
2,4-toluene diisocyanate	2.02	1.92	1.31
Cure conditions			
Cure temperature, °F	120	140	180[d]
Cure time, hr	72	72	88[d]
Measured initial mechanical properties at 77°F[e]			
Tensile strength, psi	—	112.3	152.2[d]
Elongation at maximum load, %	—	91.8	40.6[d]

[a] Column headings indicate propellant type and batch number.
[b] Polymer prepared from 2 moles of polypropylene glycol, 4 moles of 2,4-toluene diisocyanate, and 1 mole of 2,4-toluene diamine.
[c] Wetting agent by Geigy Industrial Chemicals.
[d] Undercured under these conditions; completion of cure by holding at 120°F for 2 weeks gives a tensile strength of 180 to 200 psi and an elongation at maximum load of about 35 percent.
[e] Approximate strain rate 0.74/ min.

increase which was observed for any imposed strain, regardless of the time at which it occurred. The dotted line represents Poisson's ratio calculated from Equation 2 for an incompressible material, and it is seen that values for three propellants fall far below this curve and differ appreciably from each other. (It should be noted, however, that the measurements with F-40BS propellant were made at 30°C, whereas the other two propellants were tested at 25°C.) The existence of a critical strain ε_c is denied by these results, since measured values of the Poisson's ratio deviate from the theoretical curve even at the smallest strain. Thus, it seems that dewetting of oxidizer particles is a continuous process which begins as soon as some strain is applied and is accelerated as the strain is increased.

Poisson's ratio decreases continuously over the range of strains studied (5 to 35%). While the strain rate at which the propellant rings were extended cannot be known accurately, it is at least 10 times greater than the rate normally used in tensile testing. Thus, it was not surprising to find that the X-535 propellant broke when stretched rapidly to 35 percent. In fact, both the F-40BS and the X-535 propellant rings broke at some lower strains while being held there for varying periods of time. The X-300 propellant was not tested beyond 20 percent strain because only four bubble-free rings could be obtained from the cast tube.

In order to test the possible advantages of other definitions of Poisson's ratio (Tab 1), the data were plotted as V/V_0 vs α and also as log V/V_0 vs log α. The latter plots are decidedly nonlinear, so that $d \ln (V/V_0)/d \ln \alpha$ is not constant and ν_3 cannot be readily calculated. Thus, the use of Poisson's ratio defined in terms of Hencky strains shows no advantage for propellants, in contrast with previous work on glass bead/polyvinyl chloride composites (Ref 5). There is no conflict in the conclusions, however, since the composites could be extended to much greater elongations than propellants. At low strains, the composites were also found to pass through a nonlinear transition region. The plots of V/V_0 vs α can be approximated by straight lines at strains greater than 10 percent and ν_2 can thus be calculated. The values differ greatly from ν_3, as expected.

C. Influence of Strain Upon Burning Rate

A knowledge of the effect of strain on the burning rate of composite solid propellants would provide a useful parameter for predicting the internal ballistics of large solid propellant motors. Two types of uniaxial tensile strain are convenient to employ in evaluating the influence of strain upon burning rate; these are strains normal to or parallel to the burning surface.

1. Equipment and Procedure

a. Straining/burning device. A device has been constructed to permit straining a propellant specimen normal to the direction of burning; the specimen is then burned under pressure to yield burning rate. The device (Fig 11) consists of two 5-inch diameter, 6-inch long steel cylinders connected by a 1-inch pipe. One chamber contains the propellant specimen and a stress-applying shaft which enters the top of the cylinder through an O-ring seal. The second chamber contains a high pressure nitrogen supply, a high pressure bleed valve, a dial type pressure gage, and a Statham pressure transducer; the bleed system is used to control pressure and remove hot combustion gases out of the specimen cylinder.

b. Specimen. The propellant specimen is a 2.0 inch by 1.5 inch rectangle cut from an 0.25 inch thick slab. The specimen is restricted on five sides with two clamps held

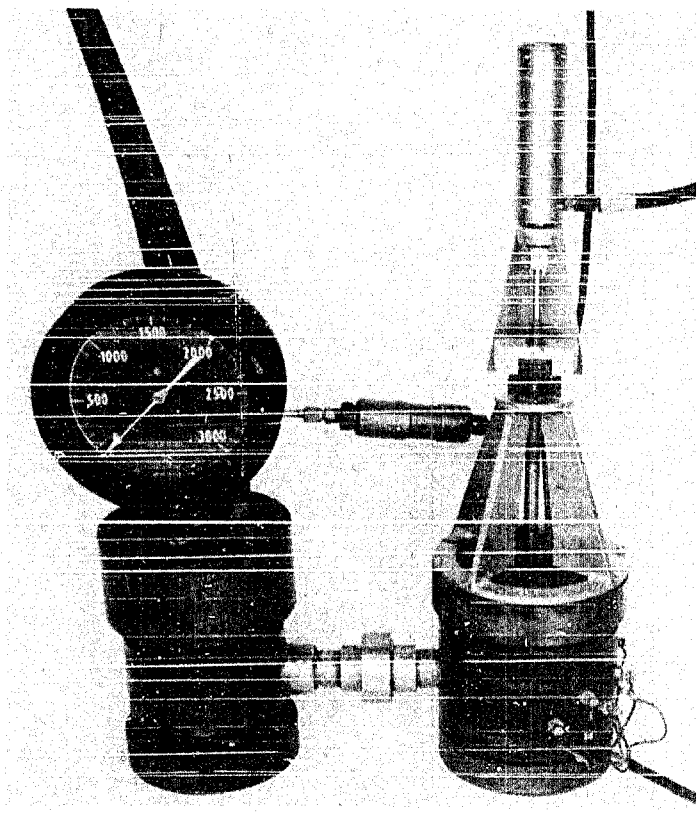

Figure 11. Two-cylinder straining device for burning-rate measurements

in place by the restrictor (Fig 12), and is attached to the bottom of the specimen cylinder and to the shaft entering the top of the cylinder. Strain on the specimen is achieved by axial displacement of the shaft; the displacement is limited by a nut threaded on the top of the shaft. A pneumatic cylinder, attached to the top of the shaft, is used to displace the shaft. After the specimen is strained and pressurized, it is ignited with a hot wire igniter.

c. Burning rate measurement. Burning rate is measured by placing wires through the propellant such that three pairs of wires are each one inch apart in the direction of burning. An electrical potential is placed across each wire and measured on an oscillograph. As the propellant burns, the wires are consumed; thus, the oscillograph record permits calculation of propellant burning rate. The pressure transducer signal is recorded on the same oscillograph record.

d. Propellant. The propellant used for these studies was an ammonium perchlorate-polyurethane type, chosen to provide good elongation properties. The tensile properties of the formulation at 77°F and a strain rate of 0.74/min are: elongation at maximum stress, $\epsilon_m = 0.550$; elongation at break, $\epsilon_b = 1.756$; and stress at maximum strain, $S_m = 41.0$ psi.

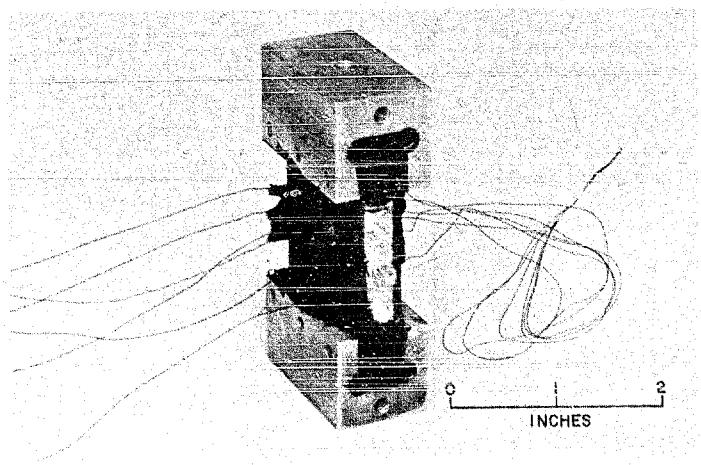

Figure 12. Propellant specimen with holding fixtures and monitoring wires

2. Preliminary Results

Some preliminary data are given (Tab 3) in which propellant burning was initiated approximately 12 seconds after application of pressure and strain. The data were taken at nominal pressures of 600, 800, 1000, and 1200 psi. The standard deviation of burning rate at 0 and 2.5 percent strain shows good reproducibility within

Table 3. Burning rate of propellant strained parallel to burning surface

Strain, %	Specimen No.	Average pressure, psia	Burning rates within specimen, in./sec			Standard deviation within specimen (±)	Average burning rate, in./sec (all data)	Standard deviation, (±; all data)
			Wire No. 1	Wire No. 2	Wire No. 3			
0	1	1210	0.292	0.273	0.299	0.013	0.278	0.014
	2	1230	0.262	0.267	0.274	0.006		
2.5	1	1230	0.304	0.299	0.300	0.003	0.299	0.005
	2	1240	0.299	0.300	0.289	0.006		
0	1	1000	0.259	0.257	0.272	0.008	0.268	0.013
	2	990	0.292	0.264	0.272	0.015		
	3	1002	0.274	0.257	0.260	0.013		
2.5	1	1000	0.271	0.272	0.275	0.003	0.275	0.007
	2	1000	0.288	0.267	0.279	0.015		
5	1	1010	0.334	0.280	0.268	0.050	0.291	0.026
	2	998	0.300	0.264	0.298	0.029		
10	1	1012	0.289	0.295	0.305	0.011	0.313	0.024
	2	1015	0.326	0.355	0.306	0.035		
0	1	800	0.251	0.236	0.241	0.008	0.246	0.007
	2	805	—	0.251	0.249	0.001		
2.5	1	815	0.256	0.256	0.259	0.002	0.256	0.004
	2	803	0.251	0.251	0.261	0.006		
5	1	810	0.271	0.271	0.276	0.003	0.270	0.009
	2	813	0.282	0.264	0.256	0.013		
0	1	605	0.234	0.240	0.238	0.003	0.237	0.003
2.5	1	625	0.265	0.241	0.241	0.014	0.244	0.011
	2	587	0.242	0.241	0.234	0.004		
5	1	585	0.238	0.247	0.254	0.008	0.249	0.009
	2	595	0.248	0.242	0.264	0.011		

and between specimens. As strain was increased to 5 and 10 percent, the spread of data was much greater, particularly within specimens; this indicates that as over-all strain increases, a nonuniformity of strain may develop within the specimen. The measurement of three burning rates in each specimen should tend to average-out the nonuniform strain effect. A distinct increase in burning rate is produced by strain (Fig 13).

Composite polyurethane propellants show evidence of forming a liquid phase at the propellant burning surface; this could produce a strain–burning rate relationship peculiar to the propellant system and should be compared with a propellant known to burn with a dry surface.

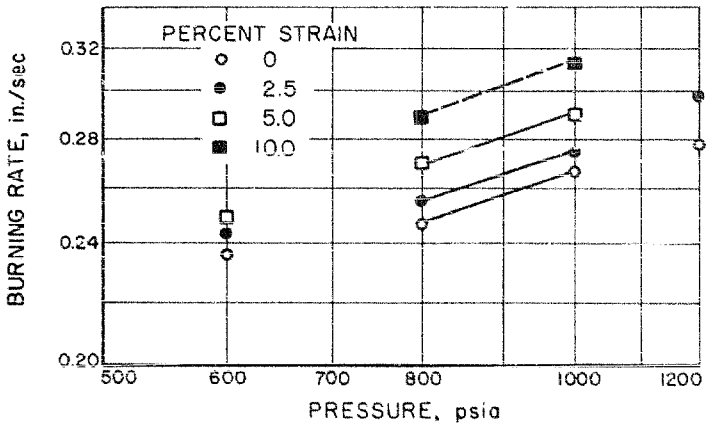

Figure 13. Effect of strain on burning rate

II. Bibliography

A. Radiography

GAMMA RAY SOURCES AND TECHNIQUES FOR GAMMA RAY RADIOGRAPHY
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February 26, 1953
Naval Ordnance Lab, White Oak, Md.
NAVORD 2666, 38 pp
ASTIA AD-19,207

A general review of radioisotopes used as radiation sources for industrial radiography is given along with production methods, measurement units, standards, and nomenclature commonly associated with their use. The specific characteristics of commercially available radiation sources are discussed as well as elements expected to be available in the near future. Particular attention has been paid to the characteristics of various isotopes which determine their suitability for specific types of radiographic inspection. Complete information for the purpose of obtaining optimum radiographs for various object thicknesses and materials is provided. Also, information is provided for the safe handling, storage, and shipping of radioisotopes of various energies.

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Massachusetts Institute of Technology, High Voltage Research Lab, Cambridge
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DA 19-020-ORD-2023
ASTIA AD-54,504 (See also AD-49,533, AD-49,533a)

The direct picture-bearing radiation transmitted through steel and the total transmitted radiation including scatter were measured for thick-target X-rays produced at 2, 3, 5, and 6 megavolts. The radiographic information derived from this data included the direct-beam absorption coefficient μ_D, the total-beam absorption coefficient μ for a 25- by 25-cm field, the ratio of scattered-to-direct radiation as a function of steel thickness, the scattering factor k which is the slope of this curve, and the figure of merit k/μ which influences radiographic sensitivity.

The radiographic latitude and sensitivity were determined, and an exposure chart was constructed for Eastman Type A industrial X-ray film over the 2- to

6-megavolt X-ray range. This voltage range yielded an increase in latitude and a small improvement in sensitivity, in addition to the expected large increase in speed and maximum steel thickness which can be radiographed. Further substantial increases in X-ray voltage resulted in increased range of secondary electrons, a diminution of available field size, and a slow increase of absorption coefficient due to pair production. No significant improvement in sensitivity was predicted for X-ray voltages beyond 4 or 5 megavolts.

DETERMINATION OF INHIBITOR THICKNESS ON 2.0-INCH GIMLET PROPELLANT GRAINS BY RADIOGRAPHY

Bujes, J. I.
April 8, 1955
Naval Ordnance Test Station, China Lake, Calif.
NOTS 1048, NAVORD 3446, 22 pp
ASTIA AD-69,103

Several methods of determining the inhibitor thickness extruded onto propellant grains were evaluated and briefly described. The radiographic method was the most applicable for use in the experimental production plant. The radiographic method fulfills a dual purpose: determining the inhibitor thickness at a glance and facilitating assessment of the grain according to the radiographic specifications. The feasibility of the method was demonstrated on reproduced X-ray films and photographs of the magnified image projected on the screen of a densitometer comparator. Detailed theoretical aspects and experimental results of the radiographic method were discussed.

SOME ASPECTS OF SOLID PROPELLANT RADIOGRAPHY (U)

Waite, G. A. C.
March 1956
Royal Aircraft Establishment, Farnborough, Great Britain
TR RPD 143, 11 pp (CONFIDENTIAL)
ASTIA AD-100,454 (See also AD-30,917)

The occurrence of an unacceptable proportion of failures during firing trials of certain solid propellant rocket charges made it necessary to carry out complete radiographic inspection of a large number of such charges. This note describes the equipment and the techniques which were employed, and shows illustrations of the more typical flaws which have been revealed. Some recommendations are made which may assist others contemplating similar work.

THE APPLICATION OF RADIOISOTOPES TO THE NON-DESTRUCTIVE TESTING OF FIBER GLASS REINFORCED PLASTICS

Kalinsky, J. L.; Di Lauro, S.
February 28, 1957
New York Naval Shipyard, Material Lab, Brooklyn
Project 5319-14
ASTIA AD-129,396

A nondestructive method, based on neutron absorption techniques, was devised to measure the amount and uniformity of the glass fiber content in reinforced plastics. The technique is apparently suitable for use in the field inspection of structures which permit the use of a neutron source and counter on opposite sides of the member being examined. With a suitable modification of the geometry, the method may provide a quantitative measure of resin content and thus yield a direct indication of the glass fiber to resin ratio.

The method is based on the relatively high absorption of slow neutrons by the boron atoms (B^{10} fraction) of the Type E borosilicate fiber glass used in fabricating the laminate. Results with a 15-mc, partially moderated RaD–Be neutron source and a B^{10}-enriched, BF_3-filled proportional counter demonstrated that the absorption of the neutron flux obeys an approximate exponential function of the glass content per unit cross section of the plastic laminate. A plot of logarithmically transmitted neutron intensity vs glass content results in essentially a linear relationship up to 0.53 cm of glass, independent of the type of weave or fabrication of the fiber glass reinforcement. The derived empirical relationships provide reliable glass assays with an average deviation of 5 percent. The technique can also be applied to determine nondestructively the H content of plastic materials composed of C, H, O, and N.

RECENT DEVELOPMENTS IN INDUSTRIAL RADIOGRAPHY: FLUOROSCOPY AND SCREEN-INTENSIFICATION PROCESSES

Halmshaw, R.
"Progress in Non-Destructive Testing," Vol 1, pp 3–31, 1958

The experimental work on the sensitivity limits attainable with fluoroscopy and with one form of image intensifier are summarized together with the factors limiting the attainable performance. The limitations on sensitivity in both fluoroscopy and image-intensifier systems, im-

posed by the effects of quantum fluctuations in the X-ray beam, are discussed. The likelihood of future progress in the field are also presented.

ASSESSMENT OF 5 mev LINEAR ACCELERATOR. PART 2. DATA FOR PRACTICAL RADIOGRAPHY

Pollitt, C. G.; Durant, R. L.; Vincent, B. J.
April 1959
Armament Research and Development Establishment, Great Britain
ARDE Memo (MX)
ASTIA AD-215,788

Exposure curves and optimum conditions for radiography with the linear accelerator are given. Wire penetrameter sensitivity curves and comparison with the sensitivities obtained with other X-ray energies are also given; although not an accurate assessment of flaw sensitivity, these data give some indication of the minimum size defect that is likely to be found under a given set of conditions. The various causes of image spread (unsharpness) are dealt with at some length, and it is shown that they are significant but not large enough to be of any serious significance when operating under the best conditions.

The linear accelerator is particularly useful in the examination of steel thicknesses ranging from 4 to 11 inches where its sensitivity is equal to that of higher energy betatrons while possessing the advantages of greater film coverage and a greatly reduced exposure time, both of which are very important factors in practical radiography. In general, it is shown that the 5 mev linear accelerator is a machine of great potentiality, particularly for the radiography of large section specimens and specimens containing a moderate (up to 2 in. steel) change in section.

THE RADIOLOGICAL EXAMINATION OF LARGE, METAL CASED, PLASTIC ROCKET MOTORS BY GAMMA RAYS (U)

Waite, G. A. C.
August 1959
Rocket Propulsion Establishment, Great Britain
RPE TM 193 (CONFIDENTIAL)
ASTIA AD-315,748

A technique is given for the radiographic examination of large, metal cased, plastic rocket motors. A device is described which will centralize the gamma ray source on the axis of the motor, so that the examination may take place with the motor horizontal.

RADIOGRAPHIC TECHNIQUES FOR LARGE PROPELLANT THICKNESSES

Holloway, J. A.; Polansky, D; Criscuolo, E. L.
February 15, 1960
Naval Ordnance Lab, White Oak, Md.
NAVORD 6813 (CONFIDENTIAL)
SPIA File No. S60-763

This report presents radiographic techniques for the inspection of large propellant thicknesses using the 2000 kvp, 10 mev, and 22 mev X-ray machines. The experimental data are given in the form of absorption and technique curves. Various lead screen and filter combinations are investigated. The optimum technique is evaluated in terms of the maximum sensitivity obtainable at each energy within practical exposure limits. An application of the data is illustrated by inspection of the X250 second stage *Polaris* motor.

RADIOGRAPHY OF CAST HIGH EXPLOSIVES

Youshaw, R. A.; Maddy, W. R.; Criscuolo, E. L.
March 15, 1960
Naval Ordnance Lab, White Oak, Md.
NAVORD 6806
ASTIA AD-236,687

This study of the X-ray absorption characteristics of cast explosives revealed a similarity between the explosives which contain aluminum as well as between those which do not contain aluminum. Comp B and H-6 were found to be representative of these two groups. It was determined that the explosives which contain aluminum have a greater absorption for X-rays than those which do not. Sensitivities better than 2 percent were obtained with all thicknesses of explosive materials at each of the X-ray energies considered. Sensitivities of ½ percent were obtained in the thickness range of 8 to 17 inches at 2000 kvp, with the greatest sensitivities being obtained at film densities between 3.0 and 4.0.

The addition of steel walls to the explosive materials degrades the radiographic sensitivity. However, sensitivities better than 1 percent were still obtained at 2000 kvp for all thicknesses of explosive between 3½ and 17 inches with ¾ inch of steel added to each side of the explosive. Exposure factors were derived from the experimental data which enabled a correction in the exposure time to

compensate for the attenuation by steel walls of varied thickness. Nylon was found to be suitable as a penetrameter material for cast explosives; however, the sensitivities obtained with nylon penetrameters are not in complete agreement with those obtained with explosive penetrameters.

POLARIS POWER PLANT DEVELOPMENT

Kirchner, W. R.
March 20, 1960
Aerojet-General Corp., Sacramento, Calif.
Report 3520-01M29, Under Patent Secrecy Order, (CONFIDENTIAL)
SPIA File No. S60-331

The time required for gammagraphic inspection of *Polaris* motors has been decreased by a factor of ⅓ by photographing the fore and aft heads simultaneously with a pair of Picker 1000-curie cameras. Test results indicate good correlation between X-ray inspection data and motor performance when the density-ratio technique of film analysis is used.

THEORETICAL DENSITY RATIOS AS A STANDARD FOR INTERPRETATION OF COBALT-60, 1000-CURIE GAMMAGRAPHS

Mascis, R. J.
March 25, 1960
Aerojet-General Corp., Sacramento, Calif.
SRP 202, SPIA File No. S60-310

A numerical standard has been established for the interpretation of cobalt-60, 1000-curie gammagraphs of *Polaris* production motors. The numerical value is obtained from a comparison of two film density readings taken with a densitometer in the area of the gammagraph containing the suspected defect. The resulting density ratio may be used successfully as a standard to differentiate between a normal and a defective area. The ratios presented in this report are adaptable to radiographs of *Polaris* motors inspected with a 2-mev X-ray unit. The general method of the film density ratio can be adapted to include radiographic and gammagraphic inspections at all energy levels and with all film types.

STATUS OF RESEARCH AND ENGINEERING PROJECTS

April 1, 1960
Allegany Ballistics Lab, Hercules Powder Co., Cumberland, Md.
ABL/QPR 19, December 1959–February 1960
NOrd 16640 (CONFIDENTIAL)
SPIA File No. S60-372

The nondestructive tests being used in this field are extrapolations and refinements of conventional techniques and, as such, are limited. A study is being made at the Allegany Ballistics Lab to evaluate those tests which are applicable and to develop new tests whenever necessary to provide inspection precise enough to predict reliable engine performance. Critical study of radiographic examination of solid propellant has revealed penetrameter sensitivity alone to be insufficient as an index of ability to see porosity of various types. Partial burning tests are being performed to determine whether visual pit count or radiography is the better method of deciding whether certain grades of porosity are anomalous or not. Experiments are being conducted to produce improved sensitivity insofar as detection of porosity is concerned for each energy level of X- and gamma-radiography. It is premature to state valid conclusions at this time, except to point out that radiography needs improvement to provide the desired degree of assurance.

FLUOROSCOPIC AND CINERADIOGRAPHIC EXAMINATION OF ROCKET BLAST TUBES DURING FIRING

Grund, M. V.
August 15, 1960
AVCO Corp., RAD Division, Wilmington, Mass.
TM-60-42 (CONFIDENTIAL)
AF 04(647)-258

The application of fluoroscopy and cineradiography to the evaluation of rocket blast-tube materials during firing of a hydrogen-oxygen rocket motor test facility is described. Radiographic and photographic variables as well as an analysis of the test results are discussed. A cineradiographic sequence of a test run in terms of unique information obtained and its influence on materials selection is analyzed.

ANALYSIS OF RADIOGRAPHIC TECHNIQUES

Holloway, J. A.; Polansky, D.; Criscuolo, E. L.
***Journal of the Society for Non-Destructive Testing*, Vol 18, pp 337–340, 1960**

An empirical method for determining radiographic technique by means of a time-latitude curve is introduced. The latitude factor, which is the ratio of the maximum exposure time to the minimum exposure time for a

given thickness and sensitivity, is presented for steel in the energy range of 140 kvp to 10 mev. Latitude factors as high as 14, which slowly decrease with increasing thickness, were found. The effects of scattered radiation on these curves is discussed.

A technique and sensitivity curve for steel at 250 kvp is presented. The data illustrate that with a given energy (250 kvcp) extreme thicknesses of flat plate steel (greater than 3 in.) can be radiographed at a 1 percent sensitivity level.

B. Ultrasonics

CONSTRUCTION AND REFINEMENT OF PROTOTYPE ULTRASONIC INSPECTION EQUIPMENT

Henry, E. A.
Sperry Products, Inc., Danbury, Conn.
February 17–May 31, 1956
Interim Report 1 on Phase 1, 50 pp
AF 33(600)-32131
ASTIA AD-100,845

A method and means of generation and application of ultra-short acoustic stress waves in nondestructive ultrasonic inspection has been developed using echo ranging techniques. The theory has been confirmed by experimental investigation where acoustic pulses of 0.06 μsec duration were used in contact inspection techniques to achieve a close-to-surface resolution of better than 0.005 inch of aluminum or steel. Several sheet metal laminates comprising epoxy bonded aluminum of various thickness between 0.010 and 0.125 inch were constructed and tested. In all cases both bonding surfaces and bond line were resolved. A void is readily detected as well as which side of the adhesive contains the void.

Contrary to other ultrasonic inspection systems, the new system provides simultaneous information in three domains—time, frequency and acoustic impedance—resulting in more precise evaluation of bond quality. A brief discussion of the three major factors that have limited the design of shortpulse ultrasonic inspection equipment precedes a simplified explanation of the theory of stress wave ultrasonic materials inspection. The performance of the experimental equipment is shown in a series of reflectograms. A time resolution of better than 0.03 μsec is shown.

ULTRASONIC TESTING WITH LAMB WAVES

Worlton, D. C.
September 25, 1956
General Electric Co., Hanford Atomic Products Operation, Richland, Wash.
Contract W-31-109-Eng-52

A method is described whereby many types of flaws lying close to the surface of a metal can be ultrasonically detected, regardless of the time duration of the interrogating pulses. Lamb waves are established in the metal between a flaw and the surface by an ultrasonic beam which impinges at the proper angle of incidence. A suitably positioned receiver transducer picks up the waves to reveal the flaw. In this method the usually troublesome surface echo is eliminated from the receiver by an acoustic barrier, making it well suited for routine and automatic testing. Results of applying the technique to several testing problems are discussed.

SOME STUDIES ON ULTRASONIC TESTING WITH LAMB WAVES

Mancuso, R. L.
February 25, 1957
General Electric Co., Hanford Atomic Products Operation, Richland, Wash.
OTS, HW-48754

Experimental data are given describing the Lamb wave response to different size unbonded areas in fuel elements as a function of crystal position. While conditions can be found for obtaining an amplitude response which varies with void width, the response is also found to be dependent to some extent on void area and depth below the surface. The results indicate that discriminating the size of unbonded area in fuel elements by the Lamb wave technique is less reliable than the count-rate method used in the present Hanford Bondtester. However, use of the Lamb wave method with close crystal spacing plus the count rate circuits for size discrimination can offer advantages in simplification of the Bondtester circuits.

THE ACOUSTIC PROPERTIES OF SOLID PROPELLANTS AT ULTRASONIC FREQUENCIES (U)

Cole, L. D.; Seaman, R. E.; Vernon, J. H. C.
May 1958
Explosives Research and Development Establishment, Great Britain
Report 2/R/58
(CONFIDENTIAL—MHA)

NEW ACOUSTIC METHOD OF FLAW DETECTION AND SOME RESULTS OF ITS APPLICATION
Lange, Yu V.
June 1960
Ministry of Aviation, Great Britain
TIL/T5084 (Translation from *Priborostroenie*, USSR, Vol 6, pp 23–25, 1959)
ASTIA AD-238,234

The principle of a new Russian acoustic method for flaw detection involves the measurement of the mechanical impedance at a point on the surface of the test item by means of the reaction on a transmitting element which sets up elastic vibrations in the article. The reaction method was used for testing the quality of adhesion of laminates. The basic element of the apparatus in the transmitting unit consists of two piezoelectric elements separated by a rod of sound-conducting material. The upper piezoelectric element is excited by an audiofrequency generator, and the lower element is connected to an amplifier. When the transmitting unit is pressed against the test item, a force of reaction deforms the lower element, and the signal at the amplifier is related to the mechanical impedance of the test item at the point of contact. If the quality of adhesion is good, the mechanical impedance is high. If there is a defect in the adhesive joint, the material in contact with the unit will vibrate independently of the rest of the structure and the mechanical impedance will be lower than in areas of good adhesion.

C. Miscellaneous

A CRITICAL SURVEY OF THE GENERAL LIMITATIONS OF THE NONDESTRUCTIVE TESTING FIELD
Ballard, D. W.
Journal of the Society for Non-Destructive Testing, Vol 15, pp 198–207, July–August 1957

A general survey of limitations in the field of nondestructive testing is described in detail.

NON-DESTRUCTIVE TESTING, A LITERATURE SEARCH
Smith, R. J.; Scott, T. W., Compilers
November 1957
Technical Information Service, Atomic Energy Commission, Oak Ridge, Tenn.
TID-3521, OTS
ASTIA AD-149,529

Bibliographic and availability information is given for 70 AEC reports and 18 non-AEC reports in which are described the following nondestructive testing techniques: sonic, ultrasonic, radiographic, radioautographic, fluorescent penetrant, electromagnetic, frost, eddy current, and leak testing.

EVALUATION OF NON-DESTRUCTIVE TEST METHODS FOR LAMINATED FIBER GLASS PLASTIC MATERIALS
Reilly, J. A.; Goldberg, M.
September 8, 1959
New York Naval Shipyard, Material Lab, Brooklyn
Final Report, Project 5761-1
ASTIA AD-226,999 (See also AD-226,066L)

Due to the increasing use of glass laminated plastic products by the Navy in the fabrication of large structural items such as boat hulls, simple and rapid nondestructive test methods are required to determine the presence of internal structural defects such as voids, resin-rich areas, and delaminations. The purpose of the investigation was to develop practical laboratory and field tests for this type of examination. Several methods were evaluated, based on the development of visual flaw patterns that result from variations in thermal conductivity through the item tested. The evaluations were conducted on a bench scale, and where practicable, full scale in-plant tests were conducted. A method based on the use of thermosensitive decals in conjunction with an infrared heat source was selected as potentially suitable, within defined limits. Recommendations were made for further field evaluation of the method.

(NO TITLE)
October 31–December 1959
Army Rocket and Guided Missile Agency, Huntsville, Ala.
ARGMA TR IDIR-2 (CONFIDENTIAL)
ASTIA AD-313,730 (See also AD-312,047)

Subjects covered are: igniter research; X-ray fluorescence analysis of polysulfide–ammonium-perchlorate propellants; plastisol nitrocellulose propellant; analyses of pyrotechnic compositions; aging program for *Nike-Hercules* sustainer motor (XM30); propellant combustion

product analysis; high energy fuels—nitrogen fluorine chemistry; ballistic bomb method for the evaluation of new propellants and propellant ingredients; radio frequency energy hazards; a preliminary study of the use of a variable-throat nozzle for a flight vehicle; and solid-propellant safety classification tests.

ENGINEERING CONSULTING SERVICES FOR GAMMA SCINTILLATION EQUIPMENT

Stephens, W. E.; Egli, F. W.; Hankowsky, J. D.; Benenson, H.
February 24, 1960
Aerojet-General Nucleonics, San Ramon, Calif.
Report AN-159, SPIA File No. S60-250

Aerojet-General Nucleonics is a consultant to Aerojet-General Corp. for further development of the gamma scintillation inspection devices for large rocket motors. The major problems associated with the stationary and portable gamma scintillation inspection devices have been resolved and are discussed in this report. The main objectives reached were: (1) the development of new techniques to reduce inspection time; (2) the development of a workable data-analysis procedure; (3) the verification of separation calibration data; (4) the investigation of high gamma background during second stage motor inspection; (5) the training of operators, mechanics, and inspectors in radiological safety and the use of radiological survey instruments; and (6) the modification of the IBM gang punch for blank column and double punch detection.

This report also includes a summary of the modifications and acceptance tests performed on portable interface bond detector No. 2 (PIBD) to insure the mechanical and electronic reliability of the gamma scintillation unit. The results of the above work show that a new inspection and data analysis technique, utilizing analog measuring and recording equipment, should be incorporated into the stationary and portable separation inspection devices. This data analysis technique will reduce the inspection and data analysis time without sacrificing separation detection sensitivity. Additional refinements meriting further investigation are also discussed.

REPORT ON NON-DESTRUCTIVE TEST METHODS USED ON POLARIS CHAMBERS AND CLOSURES

Cantor, B. R.
March 30, 1960
Naval Weapons Lab, Computation and Analysis Lab, Dahlgren, Va.
TM 115, SPIA File No. S60-432

Replies to a questionnaire received from various case fabricators on nondestructive testing of weld areas in *Polaris* motor cases are summarized and analyzed. The techniques discussed are quality control of sheets, heads and forgings, X-ray, magnetic particle inspection, ultrasonics, and dye penetrants. Recommendations are made for the improvement of techniques where necessary.

RESEARCH AND DEVELOPMENT IN SOLID-PROPELLANT ROCKETRY AND RELATED FIELDS (U)

Grover, J. H.; Wilson, J. H.; et al
February 1960
Atlantic Research Corp., Alexandria, Va.
QPR39, October 1–December, 1959, 106 pp
NOrd-15536, NOrd-18759 and NOrd-18771
(CONFIDENTIAL)
ASTIA AD-316,304

HIGH TEMPERATURE MATERIALS DEVELOPMENT

April 27, May 26, and June 30, 1960
Raytheon Co., Bedford, Mass.
Report S-150, March 1960, SPIA File No. S60-615
Report S-163, April 1960, SPIA File No. S60-705
PR S-176, May 1960, SPIA File No. S60-930
NOrd 19135 (CONFIDENTIAL)

X-ray techniques are presented that will show delaminations in free-standing pyrographite but not in pyrographite surfaces on graphite. Ultrasonic techniques are also discussed.

DEVELOPMENT AND QUALIFICATION OF A DUAL-THRUST ROCKET MOTOR FOR THE *TARTAR* MISSILE (U)

(Period covered: January 16–April 15, 1960)
Coppe, R. G.; Harvey, R. F.
May 31, 1960
Aerojet-General Corp., Azusa, Calif.
R-0118-01Q-7, NOrd 17821 (CONFIDENTIAL)

Eight full-scale basic *Tartar* motors containing ANP-2830BI Mod 0 booster propellant and ANP-2593 Mod 6 sustainer propellant were conditioned and fired during this report period. Six firings were completely successful. A faulty ceramic lining caused the exit cone to burn

through at 2 seconds during the seventh firing. The eighth motor failed at 10 seconds because of chamber overheating. A redesigned thrust neutralizer was tested and specified thrust limit achieved. Results of an investigation made to establish a nondestructive testing method for determining cracks in graphite throat inserts showed that an eddy-current inspection technique is suitable. Ultrasonic testing was tentatively adopted for determining chamber-to-liner bond integrity.

DEVELOPMENT OF A DUAL-THRUST ROCKET MOTOR FOR THE *HAWK* MISSILE

Mertens, R.; Browning, R.
Aerojet-General Corp., Sacramento, Calif.
L1914-47 (May 16–June 15, 1960),
L1914-48 (June 16–July 15, 1960)
DA-04-495-ORD-811, DA-04-495-ORD-811, Under Patent Secrecy Order, (CONFIDENTIAL)

During the qualification tests, ultrasonic and gammagraphic inspections were made on motors. It was concluded: that ultrasonic inspection will reliably detect liner-to-case bond conditions in the *Hawk* motor; that gammagraphic inspection will detect liner-insulation separation at the center wing ring; that liner-propellant bonds are excellent; that there has been no cracking or deterioration of either booster or sustainer propellants and that the areas of overheating coincided with areas of unbonding. A total of 762 motors were ultrasonically inspected at Red River Arsenal and divided into acceptable, suspect, and high-probability-of-overheating categories – 258 fell into the acceptable category with relatively little or no unbonded condition.

POLARIS PROPULSION PROGRAM

Zimmerman, C. A.
June 1960
JANAF Solid Propellant Group, Vol I, pp 33–61, SPIA
Bulletin of the 16th Meeting (CONFIDENTIAL)

An extensive propellant safety and reliability program is discussed that provides sensitivity and quality control data for A1, A2, and advanced propellants. A gammagraphic inspection facility is shown to provide rapid and complete inspection of first and second stage motors.

MINUTES OF THE TWELFTH NON-DESTRUCTIVE TEST COMMITTEE MEETING OF 14–15 JULY 1960

July 1960
Navy Dept., Bureau of Ordnance, Special Projects Office, Washington, D. C.
Meetings (CONFIDENTIAL)

Work accomplished in the development of solid rocket motor nondestructive inspection techniques as reported at the twelfth meeting of the Non-Destructive Test Committee, July, 1960, is presented.

DEVELOPMENT AND QUALIFICATION OF HIGH-PERFORMANCE, CASE-BONDED SOLID-PROPELLANT ROCKET MOTORS

(Period covered: April–June 1960)
July 29, 1960
Rocketdyne, Canoga Park, Calif.
R-4014-4 (CONFIDENTIAL)
AF 33(600)-37836, S/A No. 3

The testing of the motors in the prequalification phase of the 2-KS-10,650 rocket motor, prior to approval of the model specification, is discussed. The qualification phase of the 8-KS-2450 rocket motor and a discussion of the assembly for the 45-KS-18,000 rocket motor is presented as well as the progress made in the prequalification phase of the 1.2-KS-8600 rocket motor.

NONDESTRUCTIVE TESTING OF HARDWARE FOR SOLID ROCKET MOTORS

Joseph, S. M.
***Journal of the Society for Non-Destructive Testing*, Vol 18, pp 315–318, 1960**

The various methods used in the nondestructive testing of hardware for solid rocket motors are described, including tests used in production and those employed in development programs. The hardware and the problems pertinent thereto fall into two basic categories: (1) low alloy thin-walled steel pressure vessels heat treated in the 220,000 psi yield strength range; and (2) exit hardware of so-called exotic materials which must withstand the tremendous heat and pressures associated with the exhaust from solid rocket motors. Problems arise even with the use of long-proven methods because of the size effect and the need for detection of minute defects. Methods used on low-alloy steel rocket chambers and closures include magnetic particle inspection of forgings and welds, and X-ray and liquid penetrant inspection of welds. Under active development is a program for ultrasonic inspection of welds. Methods employed on the refractory materials include ultrasonic, penetrant, eddy current, and filtered particle inspection.

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4. Davis, R. T., *Effects of Moisture on Storage Stability of Ammonium Perchlorate Propellants*, Bulletin of the Fourth Meeting, JANAF Solid Propellant Surveillance Panel, Publication No. SPSP/7, The Solid Propellant Information Agency, The Johns Hopkins University, Silver Spring, Maryland, November, 1959.

5. Smith, T. L., *Volume Changes and Dewetting in Glass Bead–Polyvinyl Chloride Elastomeric Composites Under Large Deformations*, Progress Report No. 30-2, Jet Propulsion Laboratory, Pasadena, California, January 2, 1959.